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Adiabatic Compression Sensitivity of AF-M315E

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The Air Force Research Laboratory developed monopropellant, AF-M315E, has been selected for demonstration under the NASA sponsored Green Propellant Infusion Mission (GPIM) program. As the propulsion system developed by Aerojet-Rocketdyne for this propellant advances in maturity, studies have been undertaken to address the knowledge gaps in the adiabatic compression sensitivity of the propellant as it relates to the system parameters for this mission. Of particular interest is the sensitivity of the propellant at elevated temperatures and the resulting system peak pressures and dynamic response characteristics. For this study, an adiabatic compression U-tube apparatus was used to determine the driving pressure threshold levels of the propellant at elevated temperatures. These tests simulate the worst-case scenario resulting from a rapid closure or opening of valves in a propellant feed line *in situ*. The results of these tests are presented as a preliminary assessment on the margin of safety for the propellant.

Nomenclature

AFRL	=	Air Force Research Laboratory
K	=	bulk modulus
GPIM	=	Green Propellant Infusion Mission
HAN	=	hydroxylammonium nitrate
γ	=	ratio of specific heats
STMD	=	Space Technology Mission Directorate
k	=	spring rate
f_n	=	undamped natural frequency
W	=	weight

I. Introduction

The recent shift of focus towards green energy and technology has spurred the development of green rocket propellants. The Air Force Research Laboratory's (AFRL) monopropellant, AF-M315E, has been selected for demonstration under the NASA sponsored Green Propellant Infusion Mission (GPIM). The GPIM program was initiated by the NASA Space Technology Mission Directorate (STMD) to complete the first on-orbit demonstration of a complete AF-M315E green propellant propulsion system [1]. This mission aims to replace the current state of the art hydrazine propulsion system, in favor of a more green system for low thrust applications. The use of the highly toxic and volatile hydrazine monopropellant necessitates a variety of expensive mission considerations including safe storage, lengthy handling and disposal procedures, and many fail-safe contingencies. Many of these concerns are rectified with the use of the high-performance AF-M315E ionic liquid monopropellant. The green monopropellant offers a 50% increase in density-I_{sp} over hydrazine in addition to drastically reducing the vapor pressure and toxicity over a wide range of temperatures and pressures. Specifically, the monopropellant yields a theoretical I_{sp} of 266 seconds and density of 1.47 g/cm^3, as opposed to the 242 seconds and 1.00 g/cm^3 of hydrazine under identical conditions. In conjunction with the decreased toxicity concerns, these higher performance

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characteristics permit a wider range of volume-limited missions with an increased duration and lower overall cost [2].

With the extensive range of missions possible for AF-M315E, it is imperative that the sensitivity and stability of the monopropellant in the operating range of the various systems are established. Of particular concern to spacecraft is the adiabatic compression sensitivity of the propellant. There is a possibility of ignition within the propellant because of the rapid compression of entrained gas bubbles within the liquid. The thermal decomposition of the monopropellant produces minute gas bubbles which can cause an explosive chain reaction if the system is exposed to mechanical shock [3]. A rapid compression may be induced by an external mechanical shock on the fuel tank or by a water hammer effect caused by the rapid closure or opening of valves. Because of these deleterious effects, the sensitivity of the propellant at elevated temperatures and the resulting dynamic response characteristics are of great concern.

The goal of the current work is to expand the knowledge base from previous experiments completed at AFRL for AF-M315E in stainless steel U-tubes at room temperature. This current study wishes to determine the threshold driving pressure initiation levels as a function of temperature in a titanium alloy; one comparable to the material used in the actual GPIM propulsion system. Table 1 shows the results of previous adiabatic compressions and the rationale for this current study. There were positive responses (explosive compressions) with 350 psi driving pressure at 25 °C and 90 °C and with 300 psi driving pressure at 100 °C. Due to these results and a necessity to study temperature-dependent effects, the driving pressures were decreased to 300 psi at 25 ºC, 60 ºC, and 90 ºC, and to 250 psi at 100 ºC. These experimental parameters cover a wide range of operating conditions the propellant may experience on-orbit during the GPIM mission. The elevated temperatures may result from extended heat soak back from preheating the catalyst bed, while the driving pressures are comparable to those used in the actual propellant feed system. This comprehensive analysis of the dynamic response of the propellant is crucial to the mission as the results may dictate the final propulsion system design.

Table 1. Summary of previous AF-M315E adiabatic compressions.

Temperature [°C]	Pressure [psi]	Positive	Negative
25	350	2	8
25	400	3	1
25	500	1	0
25	1500	1	0
90	350	1	0
90	400	1	0
100	300	1	0
100	400	1	0

AF-M315E is a relatively safe propellant with the highly desirable safety properties listed in Table 2. The primary decomposition products of HAN (hydroxylammonium nitrate) based propellants such as AF-M315E are nitric acid and NOx gases. The presence of excess acids causes autocatalytic chain decomposition in the propellant. This exothermic decomposition decreases the fume-off initiation temperature of the propellant and its overall thermal stability [4]. The local temperature of the propellant may be increased from both a gradual heat soak back and a possible adiabatic compression. The resulting thermal decomposition may cause a runaway reaction that induces an explosion in a matter of milliseconds. Furthermore, the dynamic behavior of thermally damaged propellant in response to mechanical shock has not been extensively characterized; this is another prominent motivation for the current study.

Table 2. Monopropellant small-scale safety properties [5].

Characteristic	Results
Thermal stability	0.43% weight loss per 24 hours at 75 ^{O}C
Unconfined ignition response	No explosive response
Impact sensitivity [Olin Mathiesen drop weight]	60 kg-cm
Friction sensitivity [Julius Peters sliding friction]	300 N
Detonability [NOL card gap at 0 cards]	Negative
Electrostatic discharge sensitivity	Insensitive to static spark discharge (1J)
Vapor toxicity	Low hazard (No Self-Contained Breathing Apparatus)

Vapor pressure	< 0.1 torr

The safety issues associated with the storage and transfer of the liquid propellant center around the possible introduction of air bubbles, fuel vapor bubbles, and decomposition products. The high-flow rate pumping of the fuel induces drastic pressure changes in the liquid, possibly introducing vapor bubbles into the system. The bubbles form in the cavities of low pressure zones and when the liquid is exposed to a mechanical shock, these voids can implode and generate large shockwaves. The sudden collapse of a gas bubble causes a severe increase in the temperature of the surrounding liquid [6]. The mechanical work of rapid compression is then converted to thermal energy, increasing the temperature adiabatically [7]. This increase in temperature accelerates local exothermic decomposition, further increasing both temperature and gaseous decomposition products. The chain of chemical decomposition reactions may propagate an explosion if the rate of heat production is greater than that of heat loss by convection and conduction.

The temperature of the gas bubble may be estimated with the isentropic ideal gas relationship in Eq. (1) [8].

$$\frac{T_2}{T_1} = \left(\frac{P_2}{P_1}\right)^{\frac{\gamma-1}{\gamma}} \tag{1}$$

Here γ is the ratio of specific heats, T is temperature in K, and P is pressure. This equation, however, often overestimates the actual bubble temperature by assuming ideal gas behavior and constant specific heats. A more accurate adiabatic temperature may be estimated by adjusting the polytropic exponent with real gas parameters or employing viral equations of states [8, 9]. For this study, the isentropic equation is more easily employed to give a preliminary idea of the extreme temperatures possible in a true adiabatic compression.

In order to emulate adiabatic compression at various temperatures and pressures, AFRL has constructed a U-tube testing apparatus and corresponding testing method. This apparatus has been used to test the mechanical sensitivity of novel and current state-of-the-art rocket fuels and propellants. A known quantity of liquid propellant is placed in a metal U-tube and held isothermally in a preheated mixture of ethylene glycol and water. Pressure is built up in an accumulator with nitrogen gas and held behind a specified rupture disc. The propellant is then compressed with nitrogen gas and the response is characterized as positive if the U-tube ruptures and negative if it does not.

The adiabatic compression U-tube apparatus was used to determine the threshold initiation levels of the propellant at elevated temperatures and to characterize the water hammer effect. Building upon previous work at AFRL, several new parameters have been defined to thoroughly characterize the dynamic response of the tests [10]. The introduction of these well-defined parameters allows adiabatic compression tests to be better characterized as opposed to a nondescript positive or negative response.

II. Methods

Figure 1 is a detailed process and instrumentation diagram of the system used [10]. It is imperative that the entire system is placed in an isolated test cell in case of an explosive positive response. In order to maximize the response time of the test and minimize the deviation from adiabaticity, a fast acting solenoid valve and burst disc are used. The use of a burst disc instead of a standard valve gives response times on the order of 10 to 20 ms. In addition, the burst disc prevents vapors or decomposition products of the propellant from escaping back downstream into the system. The rupture discs are precision machined and certified to burst at predetermined pressures.

Nitrogen gas is pressurized to specific driving pressures into an accumulator tank above a rapid-opening valve. This valve is placed directly above the burst disc and the U-tube containing preheated propellant. The U-tube has a 0.250 inch nominal OD, 28 mL volume, and is made of a titanium 3Al2.5V alloy. A simple calculation of Barlow’s Formula [11] for this alloy, yields bursting and deformation pressures of approximately 21,100 psi and 19,300 psi, respectively. This alloy was chosen specifically for its strength and material compatibility with a wide range of propellants, including AF-M315E. This monopropellant is especially sensitive to transition metals with multiple oxidation states, but appears to be compatible with this titanium alloy.

Prior to a compression test, the U-tube is pre-cleaned with acetone and methanol and purged with nitrogen. 3 mL of propellant is then injected with a syringe into the U-tube and the test setup is reassembled. An important facet of this setup is that the propellant sits at the very bottom of the U-tube with a blanketing layer of air on either side. This is an important distinction from other setups, which instead, use straight-line vacuum evacuated tubes downstream from the rapid-opening valves. However, these systems often cannot preheat the propellant to operating temperatures, an inherent benefit of this U-tube setup.

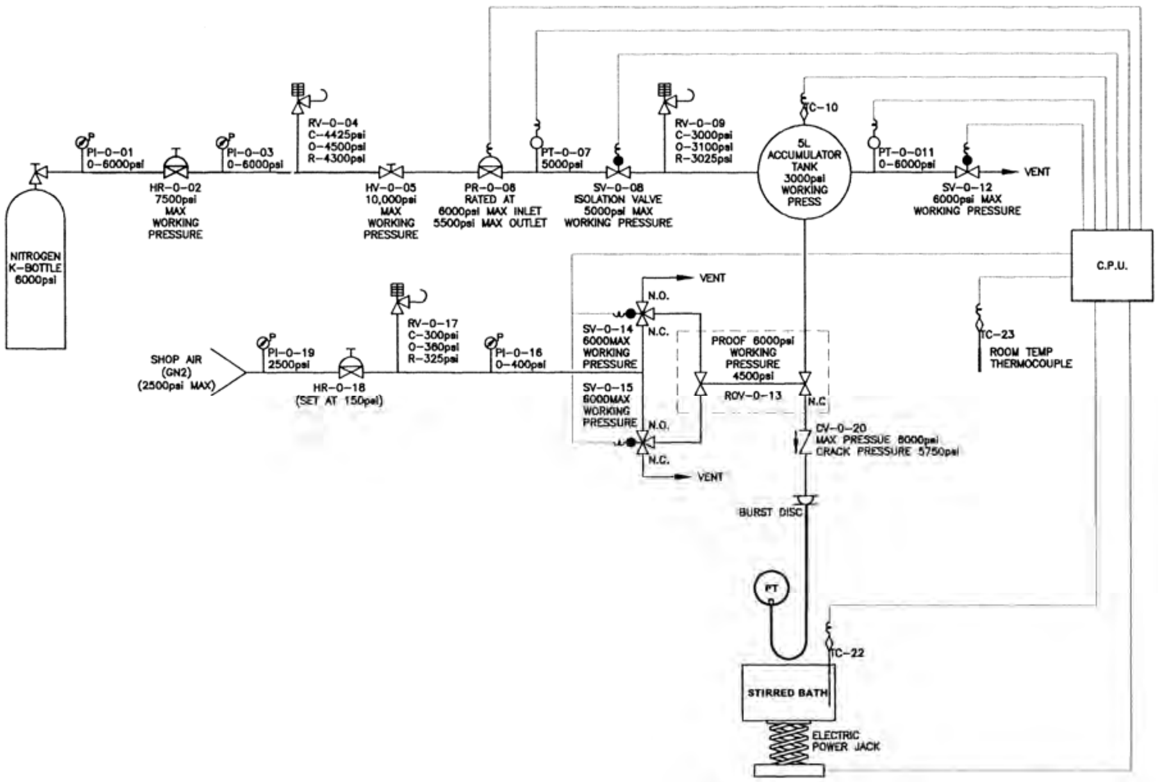

Figure 1. Process and instrumentation diagram of test facility.

After loading the propellant, the U-tube is secured on one side and closed off to a Kistler 211B1 piezoelectric pressure transducer on the other. The high sensitivity of the piezoelectric transducers makes them especially prone to any vibrations present in the system, particularly the resonance of the metal tubes after sudden shock. This problem should be addressed, to some degree, with the use of clamps and a large stainless steel plate to dissipate any major vibrations. A large preheated bath of 50:50 v/v mixture of water and ethylene glycol is then raised up against the steel plate containing ports for the U-tube. Approximately 95% of the U-tube is submerged in the temperature bath and soaked isothermally for 30 minutes before testing. This soaking period was chosen to ensure that the entirety of the propellant was heated to and held at a uniform temperature. Although the propellant reaches thermal equilibrium in a few minutes, this soaking period was selected as a conservative estimate of how long the propellant may be exposed to higher temperatures during the catalyst bed preheat.

The previous system at AFRL was retrofitted with a new high-speed data acquisition device to capture the peak pressures and large pressure oscillations [10]. Because of this, a triggering mechanism for automatic data collection has yet to be configured. In order to rectify this problem, data is collected slightly prior to valve opening. The pressure is captured for 5 seconds at 25 kHz and repeated in triplicate. This high sampling rate should prevent aliasing of the data and accurately capture the pressure traces of each test.

A. Data Analysis by MATLAB

Preliminary tests showed that the adiabatic compressions yielded large oscillations about the driving pressures. These curves were similar to an underdamped response in a 2nd order dynamic system [12]. Drawing inspiration from process and dynamics control, various terms were defined to thoroughly characterize the pressure traces obtained. The *Terminology* section below defines these terms and Figure 2 depicts the relative positions of these characteristics on a representative pressure trace.

After the data was collected by LabView, it was analyzed using a script written in MATLAB. Due to the delay between data collection and actual test, the starting time point had to be shifted accordingly. The starting time was set by calculating a differential pressure between sequential data points. Once the differential pressure was greater than a set nominal value, the data point directly preceding this was set as the start time. This start time approximated the beginning of the rapid rise in pressure caused by the compression. After the start time was set, the rest of the parameters could be determined according to the following definitions.

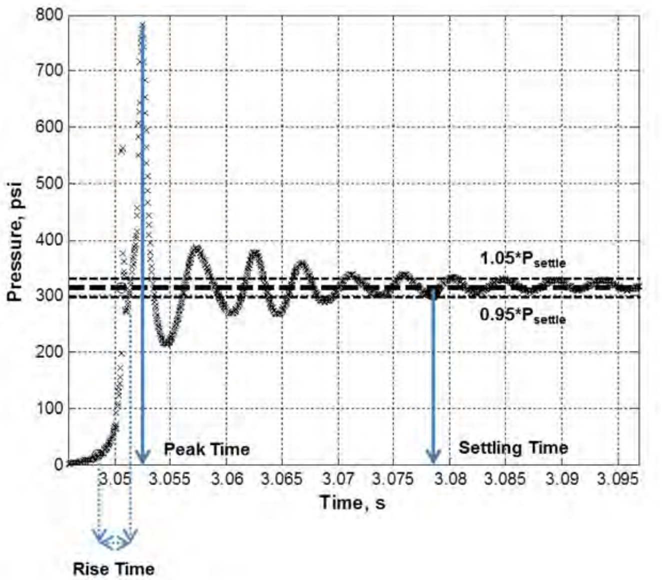

Figure 2. Sample graph with characteristics times.

B. Terminology

Max Pressure – Characteristic peak pressure of the test.
Settling Pressure – The final steady state pressure of the test. This is equal to the initial driving pressure.
Peak Time – Approximate time taken for the system to reach its peak pressure relative to starting time point.
Rise Time – Time taken for system to go from 5% to 95% of the settling pressure.
Settling Time – Time taken for pressure to stabilize within a ± 5% band of the settling pressure.
Compression or Pressurization Rate – Ratio of settling pressure to rise time.

III. Results and Discussion

A preliminary round of testing was completed on ambient water at 300 psi to serve as a baseline water hammer test. A representative sample of a water and propellant test is shown in Fig. 3A and Fig. 3B, respectively. This behavior may be modeled as a classical spring-mass system, with the slug of propellant treated as a uniform mass. The volume of gas on either side of the slug of propellant acts as springs, compressing and decompressing the propellant in the U-tube. This response has been confirmed in previous studies and is not due to an underdamped transducer [13, 14].

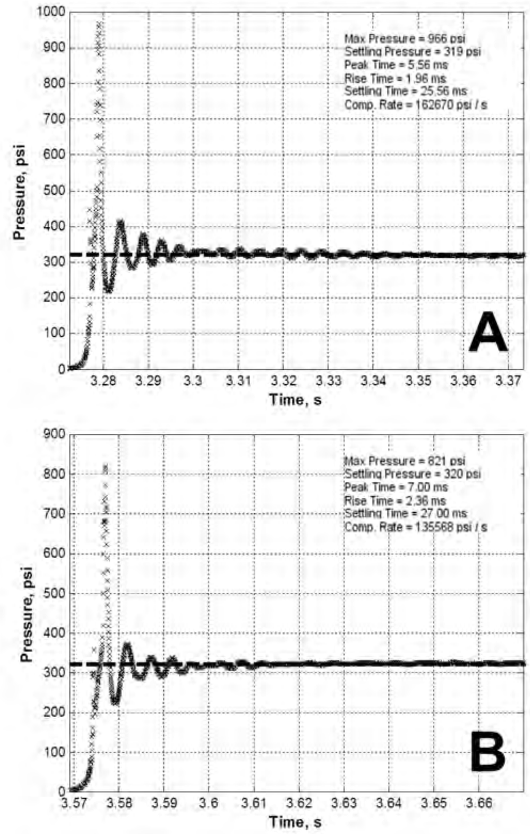

Figure 3. Representative pressure trace of a) water and b) AF-M315E at 300 psi and 25 ºC.

The large overshoots in pressures and oscillations may be partially explained by the water hammer effect intrinsic to the sudden compression of a fluid. A water hammer is a massive surge of pressure caused by suddenly stopping or starting a liquid flow [15]. The sudden compression of one side of the propellant induces shock waves, which propagate through the propellant slug and into the gas front. The shock wave does not travel through a homogenous and single phased fluid and is instead, partially dissipated by a compressible gas on either side of the U-tube. Although the results may not be adequately explained by the water hammer effect, it offers insight into the underlying processes of this particular system. The presence of these shock waves is important because of their significant contribution to the thermal and pressure profile of the tests [13]. It should be noted that the response of the system to these compressions is due to the conjugation of isentropic compression and shock effects, rather than either effect alone [16].

Both tests in Fig. 3 have similar rapidly decaying oscillatory responses and comparable calculated parameters, with the exception of a markedly different pressure trace. The decay ratios, or ratio of the primary and secondary peaks, of the water hammers were noticeably larger and had slightly smaller periods of oscillations. In effect, the oscillations experienced by the propellant decayed at a faster rate than that of the water hammer. In lieu of a simplified model of resonance in a spring-mass system, this may be caused by the difference in bulk modulus of water (2.2×10^9 Pa) and AF-M315E[4] (5.7×10^9 Pa) [17].

Equation (2) is used to calculate the undamped natural frequency, or f_n, of an ideal vibrational system. Here, f_n is in Hz, k is the spring rate in lb / in., W is weight in lb.

[4] Calculated from an estimate of sonic velocity of AF-M315E (1980 m/s) provided by NASA Goddard Space Flight Center.

$$f_n = \frac{1}{2\pi}\sqrt{\frac{k}{W}} \tag{2}$$

In order to solve the system of equations modeling the true process, a highly detailed analysis of the system must be completed to derive the corresponding damped natural frequency as a function of damping ratio. Instead, a simplified analog hydraulic system is obtained by replacing k with K, or bulk modulus [18]. By lowering the natural frequency, the overall system stability is greatly reduced. Therefore, a lower modulus equates to a lower natural frequency and subsequent overall stability of the system. This model adequately explains the results as the lower bulk modulus of water caused a more unstable and highly oscillatory response in comparison to the propellant, as shown in Fig. 3A and 3B.

Previous studies on commercial hydraulic fluid have found that raising the temperature 38 °C reduces the bulk modulus to 61% of its room temperature value. Introducing 1% air by volume also reduced the bulk modulus to 55% of its room temperature value [18]. The combination of both effects drastically reduces the bulk modulus and ultimate stability of the system. The effect of temperature on bulk modulus and compressibility may offer a partial explanation to the average results shown in Table 3 and representative samples shown in Fig. 4. Refer to Table A1 in the Appendix for a complete list of adiabatic compressions at various driving pressures and temperatures.

Table 3. Average parameters for triplicate samples.

Parameter	**Water**	**AF-M315E**			
	300 psi	**300 psi**			**250 psi**
	25 °C	25 °C	60 °C	90 °C	100 °C
Maximum Pressure [psi]	797	829	840	833	718
Settling Pressure [psi]	313	318	314	313	253
Peak Time [ms]	5.41	7.04	7.35	6.39	7.53
Rise Time [ms]	2.04	2.37	2.75	2.80	3.31
Settling Time [ms]	28.75	27.04	67.37	72.23	87.61
Compression Rate [psi / s]	153805	133916	114932	111852	76667
Est. Adiabatic Temp. [°C] [5]	--	444	526	597	568

Closer inspection of Fig. 4 reveals small discontinuities during the initial rise in pressure. This discontinuity may be attributed to the vigorous turbulence of the liquid-vapor interface upon sudden compression. The rapid compression may cause a splattering of the propellant from the liquid surface to the transducer directly prior to arrival of the main propellant slug. This “double peak” appears to be more pronounced, with a larger magnitude, at higher temperatures. This may be due to the temperature dependent viscosity and surface tension effects of the propellant. At the higher temperatures, the lower viscosity and surface tension makes it more likely for larger droplets of propellant to be ejected from the liquid-vapor interface, resulting in the larger double peak.

The average peak pressures obtained were very similar even as the temperature was increased. For all of the 300 psi tests, the average peak pressures were approximately 2.7 times their respective driving pressures. This result is significant as the peak pressures observed may dictate whether the propellant is suitable for certain propellant feed systems. Although there is no conclusive evidence that the fast pressurization and large overshoot of the propellant may cause an explosion, the sensitivity of the propellant vapors is often subject to concern [3].

AF-M315E is a thermally stable monopropellant, with the only volatiles being a small amount of water and any thermal decomposition products such as NO_x gases. The overall vapor pressure, in the absence of water, is less than 0.1 torr due to the stable nature of these liquid ionic salts. A notable aspect of this particular monopropellant is that its vapors are not particularly sensitive to combustion or ignition, even in an oxygen rich atmosphere. This may explain why the peak pressures observed are similar even as the temperature was increased. In absence of a chemical reaction, the peak pressures observed in these adiabatic compressions are a function of the driving pressure, rather than the initial temperature of the monopropellant.

[5] Assuming a constant γ = 1.42 of room temperature air.

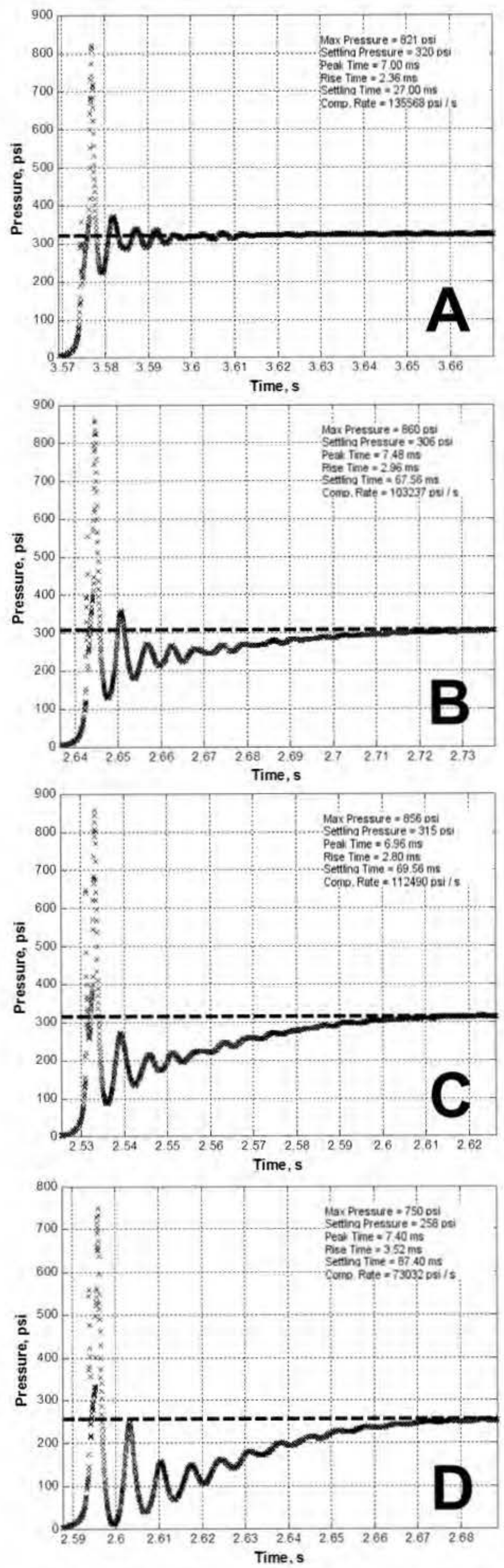

Figure 4. Adiabatic compressions at 300 psi and a) 25 °C, b) 60 °C, c) 90 °C and 250 psi at d) 100 °C.

At the elevated temperatures shown in Fig. 4A to 4D, the pressure oscillates below the settling pressure and gradually increases to the driving pressure. The pressure traces exhibited a sharp initial pressurization and slow oscillatory rise to the driving pressure. The pressure of the secondary peaks of each test was successively lower as the temperature was increased. Additionally, the ΔP between each peak and trough and the period of oscillation appeared to grow larger. This behavior is indicative of a partial decomposition of the propellant as opposed to an ignition event. Partial decomposition is theorized to occur because of the extreme liquid breakup and high rate of interfacial heat transfer [19]. The heat generated from the adiabatic compression of the propellant is quickly dissipated along the interior wall of the U-tube. Because of the large surface area for heat transfer relative to the slug of propellant, there is no point source at a high enough temperature sufficient for the propellant to ignite. Instead, certain areas of the propellant slug may have partially decomposed at the adiabatic temperatures listed in Table 3, generating small amounts of gas. The dynamic behavior exhibited may be explained by this partial decomposition and lower bulk modulus as a function of temperature.

There was a general increase in all of the characteristic times of the tests as shown in Fig. 5. The slopes of the linear regression drawn in Fig. 5A are nearly zero, indicating a very weak relationship between the temperature and rise and peak times. The settling time had a very strong temperature dependency as shown in Fig. 5B. The average settling time drastically increased from 27 to 67 ms for the 25 °C and 60 °C tests, respectively. Even though the driving pressures of the 100 °C were 50 psi lower, the characteristic times appeared to follow the general trend of the 300 psi tests as shown in Fig. 5. These linear regressions need many more data points before there is any degree of confidence in the ability to predict the characteristic times as a function of temperature. The lines plotted simply show the general trend observed, rather than serving as a predictor of response characteristics.

There was a decrease in the compression rate as the temperature was increased. This behavior was expected, as the compression rate is a function of the settling time and constant driving pressure. The compression rates observed confirm estimates from previous studies completed at the lab [10].

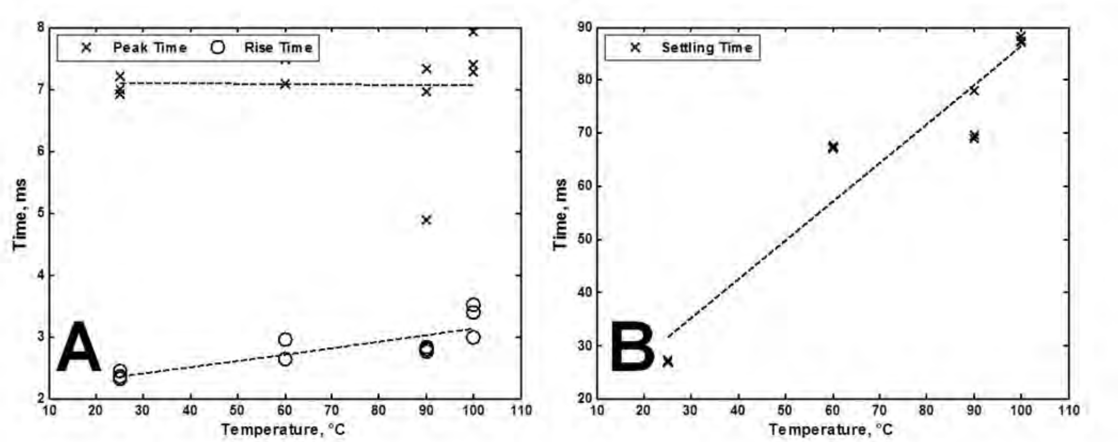

Figure 5. Calculated a) peak and rise and b) settling times for all tests.

IV. Conclusions

The U-tube apparatus developed at AFRL has demonstrated remarkable repeatability for adiabatic compression testing of monopropellants. The results of this study are valuable to anyone desiring a high performance monopropellant for use in a volume-limited application such as spacecraft. The peak pressures observed in the study help determine the strength of material required in any given system, while the characteristic times and compression rates highlight the response characteristics of the propellant at certain temperatures and driving pressures. These results are vital in the design of a safe and reliable AF-M315E propulsion system.

The detailed study of AF-M315E at various temperatures and driving pressures ensures that the propellant will perform up to spec at certain operating conditions. There is a high degree of confidence that the propellant will not experience an explosive adiabatic compression at a 300 psi driving pressure from 25 °C to 90 °C. However, at ambient temperatures, the maximum driving pressure should not exceed 350 psi to prevent a possible explosion. Although the propellant is safe up to 100 °C at 250 psi driving pressure, prolonged exposure to high temperature will initiate an irreversible thermal decomposition over time. This thermal decomposition degrades the performance of the propellant and increases the sensitivity of the propellant to other environmental factors.

As the technology readiness level of the AF-M315E propulsion system matures, future studies will be required to reassess the safety margin for possible changes in system design. There will also be a need for extended study as more potential applications for the monopropellant are found. As it stands, the propellant should perform well if the results of this study are taken into consideration when designing a dedicated AF-M315E propulsion system.

Appendix

Table A1. Comprehensive list of all AF-M315E adiabatic compressions.

Temperature [°C]	Pressure [psi]	POS	NEG
25	**300**	**0**	**19**
25	350	2	8
25	400	3	1
25	500	1	0
25	1500	1	0
60	**300**	**0**	**8**
90	**300**	**0**	**21**
90	350	1	0
90	400	1	0
100	**250**	**0**	**19**
100	300	1	0
100	400	1	0

Table A2. Response characteristics and average values for all tests.

Parameter	AF-M315E											
	25 °C, 300 psi				60 °C, 300 psi				90 °C, 300 psi			
	A	B	C	Avg	A	B	C	Avg	A	B	C	Avg
Max Pressure [psi]	807	821	859	829	860	872	790	840	856	919	724	833
Settling Pressure [psi]	316	320	317	318	306	315	323	314	315	312	312	313
Peak Time [ms]	6.92	7.00	7.20	7.04	7.48	7.08	7.48	7.35	6.96	7.32	4.88	6.39
Rise Time [ms]	2.32	2.36	2.44	2.37	2.96	2.64	2.64	2.75	2.80	2.84	2.76	2.80
Settling Time [ms]	26.92	27.00	27.20	27.04	67.56	67.08	67.48	67.37	69.56	69.12	78.00	72.23
Compression Rate [psi / s]	136148	135568	130030	133916	103237	119380	122179	114932	112489	109876	113190	111852

Parameter	AF-M315E				Waterhammer			
	100 °C, 250 psi				25 °C, 300 psi			
	A	B	C	Avg	A	B	C	Avg
Max Pressure [psi]	681	750	723	718	966	782	645	797
Settling Pressure [psi]	254	257	247	253	319	314	307	313
Peak Time [ms]	7.92	7.40	7.28	7.53	5.56	5.60	5.08	5.41
Rise Time [ms]	3.40	3.52	3.00	3.31	1.96	2.08	2.08	2.04
Settling Time [ms]	88.16	87.40	87.28	87.61	25.56	35.60	25.08	28.75
Compression Rate [psi / s]	74628	73031	82341	76667	162669	151087	147660	153805

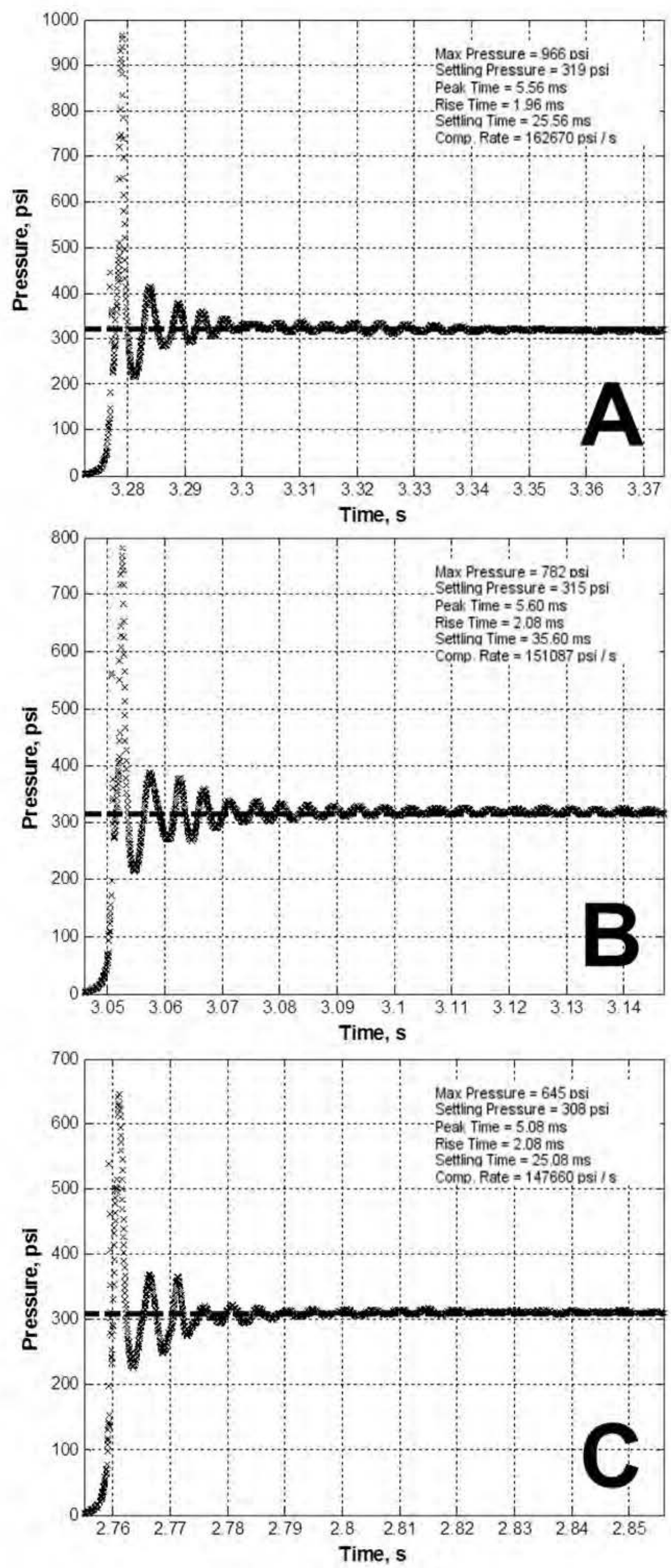

Figure A1. Triplicate adiabatic compressions for water at 25 °C and 300 psi.

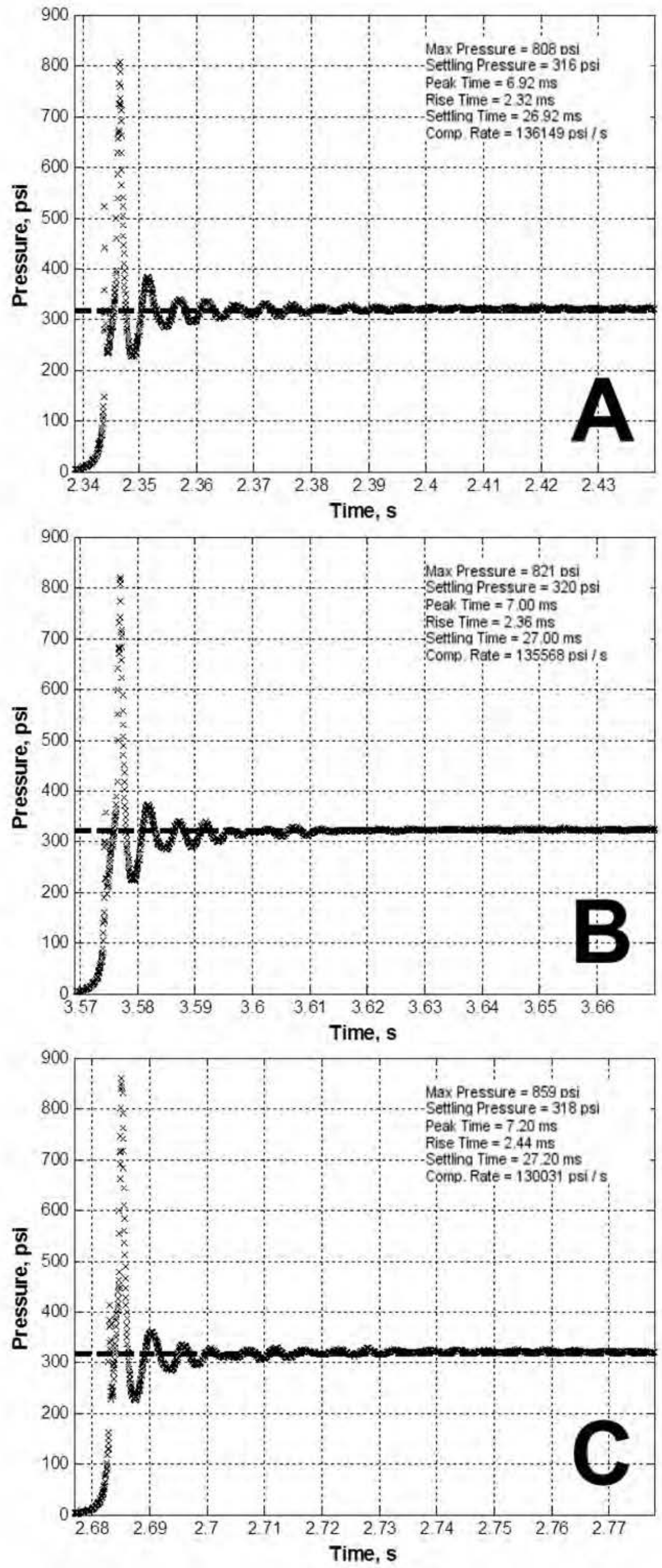

Figure A2. Triplicate adiabatic compressions for AF-M315E at 25 °C and 300 psi.

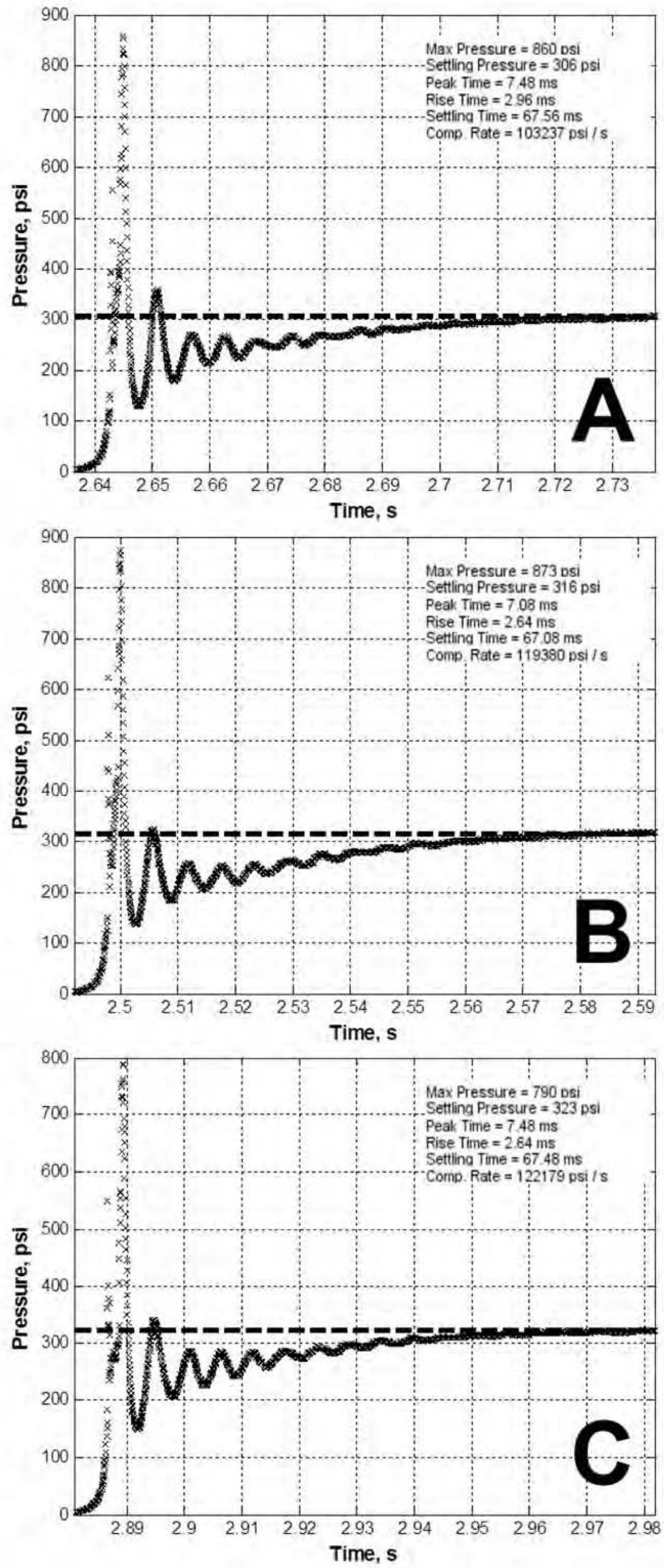

Figure A3. Triplicate adiabatic compressions for AF-M315E at 60 °C and 300 psi.

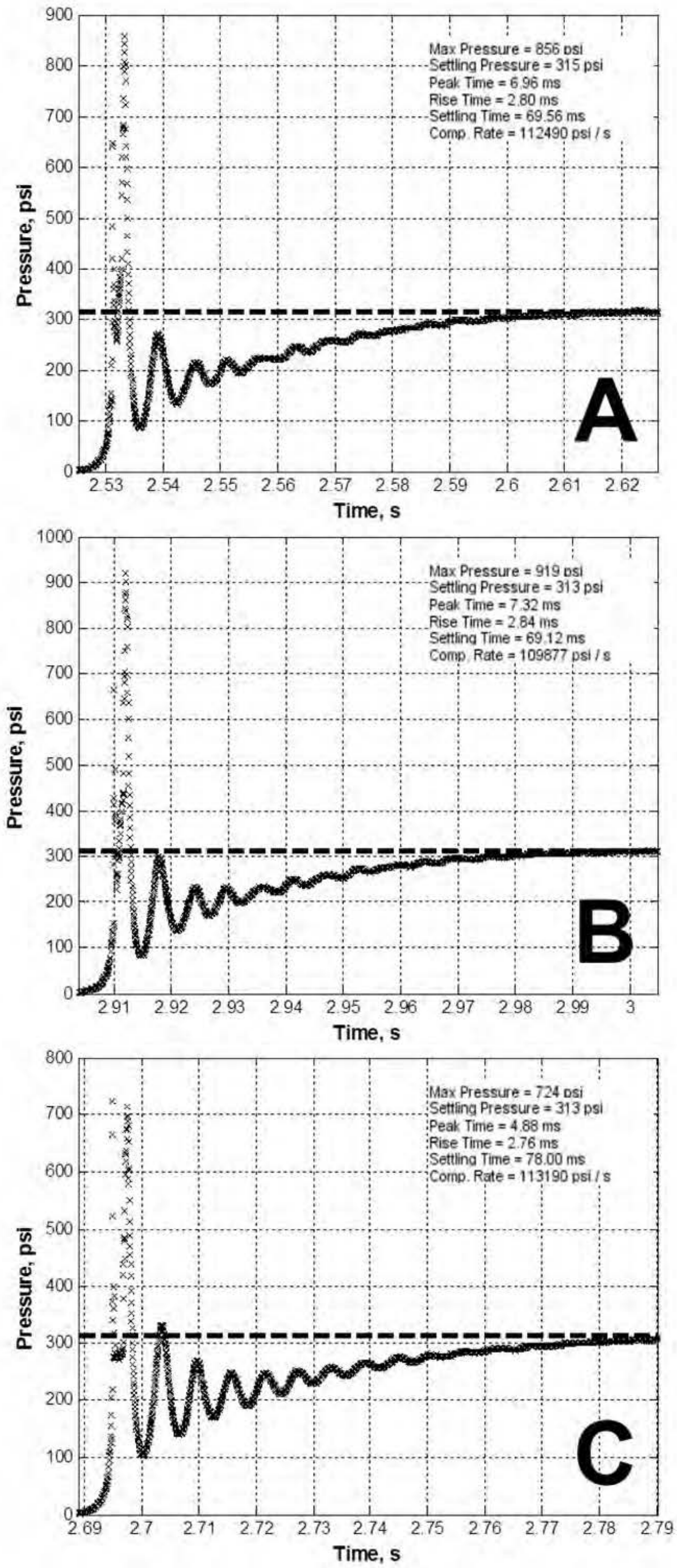

Figure A4. Triplicate adiabatic compressions for AF-M315E at 90 °C and 300 psi.

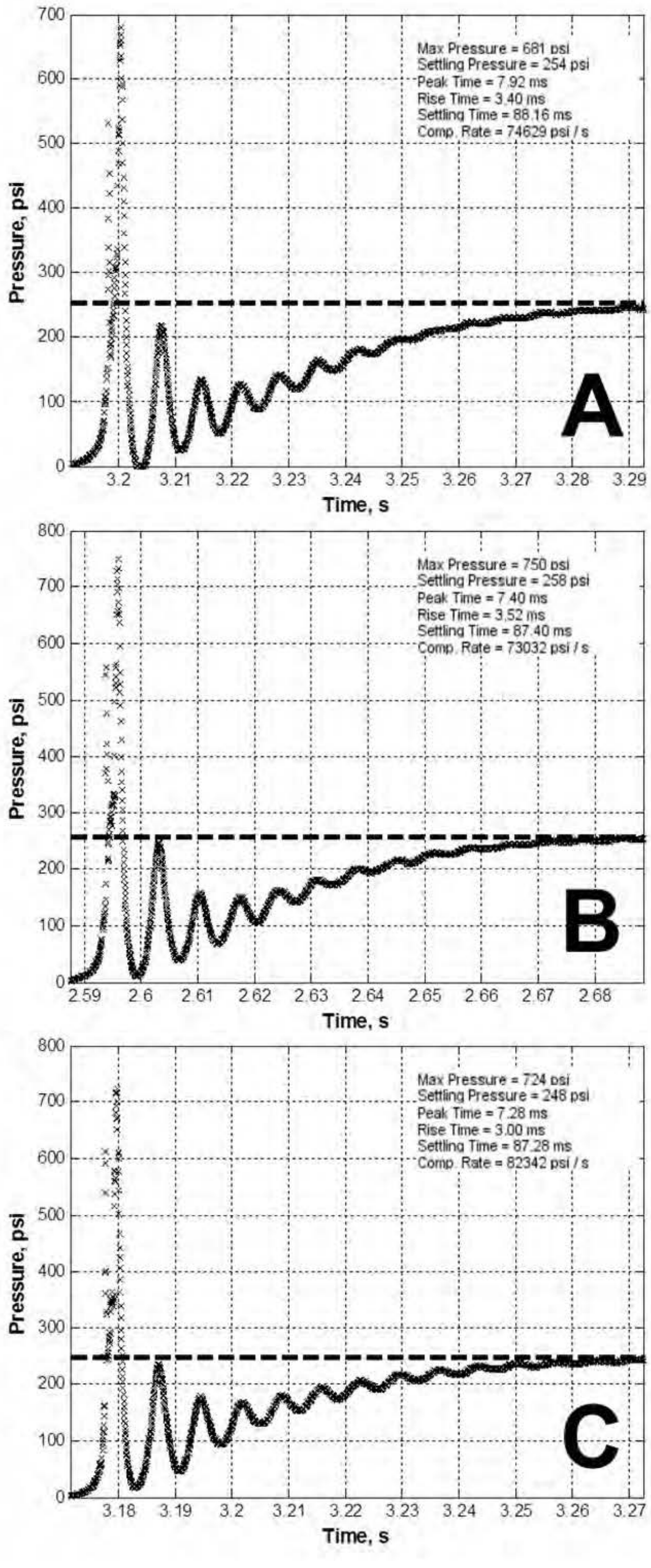

Figure A5. Triplicate adiabatic compressions for AF-M315E at 100 °C and 250 psi.

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